

<p align="center"><b>APPENDIX 15 – SEM/EDS</b></p>	<p align="center">Page 1 of 3</p>
<p align="center"><b>Division of Forensic Science</b></p> <p align="center"><b>TRACE EVIDENCE PROCEDURES MANUAL</b></p>	<p>Amendment Designator:</p>
	<p>Effective Date: 31-March-2003</p>
<p><b>15 SCANNING ELECTRON MICROSCOPE/ ENERGY DISPERSIVE X-RAY ANALYZER (SEM/EDS)</b></p> <p><b>A.</b> A service contract is kept on each instrument which includes comprehensive annual preventive maintenance visits.</p> <p><b>B.</b> On a daily basis, the calibration of the x-ray analyzer is checked and logged with an appropriate standard.</p> <p><b>C.</b> The liquid nitrogen Dewar on the x-ray detector is filled at least twice a week to insure that it does not go dry.</p> <p><b>D.</b> The in-line water filter is cleaned mid-month or as necessary.</p> <p><b>E.</b> The filament assembly is cleaned every four months or when the filament burns out. The filament is replaced every four months or as needed. Be aware of elevated temperatures when changing a filament which has been in operation.</p> <p>a. During filament replacement on the R.J. Lee or ASPEX Instrument be sure to disable the beam power supply located on the back panel of the beam supply under the bias knob.</p> <p><b>F.</b> Change apertures as indicated by stigmatism.</p> <p><b>G.</b> The mechanical vacuum pump oil is changed annually.</p> <p><b>H.</b> The resolution is checked monthly by taking a 25,000X micrograph of an appropriate standard.</p> <p><b>I.</b> Service, maintenance and other calibration data are logged and kept in the SEM laboratory.</p> <p><b>J. Zeiss/Oxford, R. J. Lee, ASPEX Daily Operations Log</b></p> <p>a. A bound log is kept for documenting the daily operations of the Zeiss DSM 940A and 960A Scanning Electron Microscopes and the Oxford eXL Energy Dispersive X-ray equipment. This log is separated into columns that include appropriate parameters and a comments section. Comments refer to filament changes, overnight runs and equipment adjustments as they arise. A similar log is kept for the R. J. Lee and ASPEX Instrument.</p> <p><b>K. SEM/EDS System Maintenance Schedule</b></p> <p>a. Fill the dewar with liquid nitrogen every Monday morning, Wednesday, and Friday afternoon.</p> <p>b. When the line pressure falls below 20 PSI, clean the filter screen.</p> <p>c. Change the mechanical pump oil every 12 months or when it becomes cloudy.</p> <p>d. Change apertures as indicated by stigmatism.</p> <p><b>L. SEM/EDS Zeiss/Oxford System Emergency Shutdown Procedures</b></p> <p>a. Turn key on link eXL computer from on to off position.</p> <p>b. Turn power switch off on front of Mitsubishi video printer.</p> <p>c. Turn power switch off on small video monitor.</p> <p>d. Turn power switch off on Epson printer.</p> <p>e. ZEISS/OXFORD SEM/EDS SYSTEMS CAN ALSO BE TURNED OFF BY THROWING THE KNIFE SWITCH ON THE WALL. THIS IS AN <b>EMERGENCY</b> SHUTDOWN PROCEDURE. DATA WILL BE LOST IF DOING AN AUTOMATED GSR RUN.</p>	

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<p><b>M. R. J. Lee Emergency Shutdown Procedures</b></p> <ul style="list-style-type: none"> <li>a. Hit Alt Esc keys from keyboard.</li> <li>b. Shut down computer in Windows mode.</li> <li>c. Turn off computer power switch on SEM console.</li> <li>d. Turn off main power rocker switch on SEM console.</li> </ul> <p><b>N. ASPEX Emergency Shutdown Procedures</b></p> <ul style="list-style-type: none"> <li>a. Turn off filament.</li> <li>b. Minimize perception software.</li> <li>c. Shutdown Windows 2000.</li> <li>d. Turn off main, power and pulse proc. toggle switches on SEM.</li> </ul> <p><b>O. QC Oxford System eXL EDS</b></p> <ul style="list-style-type: none"> <li>a. Daily Calibration <ul style="list-style-type: none"> <li>i. Use a Co standard</li> <li>ii. MICROSCOPE CONDITIONS: <div style="margin-left: 40px;"> 20 kV  20 eV/CHANNEL  25mm WD  2000 CPS  2000 X  FAST COUNTING MODE  PROCESSOR TIME 3 </div> </li> <li>iii. Run gain calibration program through ZAF-PB</li> <li>iv. Keep hard copy of resolution</li> </ul> </li> <li>b. Autocalibration, as needed <ul style="list-style-type: none"> <li>i. Use a Co standard.</li> <li>ii. Set up the microscope conditions as listed in DAILY CALIBRATION and run the AUTOCALIBRATION program.</li> </ul> </li> </ul> <p><b>P. R. J. Lee EDS System</b></p> <ul style="list-style-type: none"> <li>a. Daily Calibration <ul style="list-style-type: none"> <li>i. Use a Cu Standard</li> </ul> </li> </ul>	

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<div> <div> <div>ii.</div> <div>Do a 2 point calibration on Cu K<math>\alpha</math> and L<math>\alpha</math> lines. These peaks should be symmetrical through the peak centroid line. Working conditions are: 20kV, 15mm WD, 6000CPS or Dead Time not to exceed 40%, VFS 8000, from 0 to 10.24 KeV.</div> </div> <div> <div>iii.</div> <div>During automated GSR runs the centroids of these peaks will be monitored. The Cu K<math>\alpha</math> centroid should be 8.04 <math>\pm</math>.03 KeV. The Cu L<math>\alpha</math> centroid should be 0.93<math>\pm</math>.03 KeV. FWHM of Cu K<math>\alpha</math> will also be monitored during the automated run. It should not exceed 185 eV.</div> </div> <div> <div>iv.</div> <div>A hard copy of the data collected during automated GSR runs will be kept in a QC notebook.</div> </div> </div> <div> <div>Q.</div> <div>ASPEX EDS System</div> <div> <div>a.</div> <div>Daily Calibration</div> <div> <div>i.</div> <div>Use a Cu Standard</div> </div> <div> <div>ii.</div> <div>Do a 2 point calibration on Cu K<math>\alpha</math> and L<math>\alpha</math> lines. These peaks should be symmetrical through the peak centroid line. Working conditions are: 20kV, 15mm WD, 3000CPS or Dead Time not to exceed 45%, VFS 8000, from 0 to 10.24 KeV.</div> </div> <div> <div>iii.</div> <div>During automated GSR runs the centroids of these peaks will be monitored. The Cu K<math>\alpha</math> centroid should be 8.04 <math>\pm</math>.03 KeV. The Cu L<math>\alpha</math> centroid should be 0.93<math>\pm</math>.03 KeV. FWHM of Cu K<math>\alpha</math> will also be monitored during the automated run. It should not exceed 185 eV.</div> </div> <div> <div>iv.</div> <div>A hard copy of the data collected during automated GSR runs will be kept in a QC notebook.</div> </div> </div> </div> <div> <div>R.</div> <div>SEM/EDS GSR Kit – QC</div> <div> <div>a.</div> <div>Each box of kits from the manufacturer must have a lot number associated with it.</div> </div> <div> <div>b.</div> <div>Select 2% of the kits received from the manufacturer.</div> <div> <div>i.</div> <div>Inspect the kits to ensure all components are present.</div> </div> <div> <div>ii.</div> <div>Visually inspect the sample stubs. The stubs should be completely covered with carbon conductive tape.</div> </div> <div> <div>iii.</div> <div>Run an automated GSR analysis on one sample from each kit to ensure no GSR or contaminating heavy metal particles are present.</div> </div> <div> <div>iv.</div> <div>The QC'd kits are retained and used as negative controls for automated GSR runs.</div> </div> </div> <div> <div>c.</div> <div>Distribute the GSR kits to the Regional Laboratories via lockbox.</div> </div> <div> <div>d.</div> <div>Track the distribution of GSR kits to Law Enforcement Agencies from the Central and Regional Laboratories via FACTS.</div> </div> <div> <div>◆End</div> </div> </div>	